

PHILIP MORRIS U. S. A.

INTER - OFFICE CORRESPONDENCE

Richmond, Virginia

To: . Dr. Jane Y. Lewis

Date: January 16, 1990

From: . Jeff Sampson

Subject: . Accomplishments for 1989

I. INSTRUMENTATION, METHODS DEVELOPMENT AND IMPROVEMENT

- A. Objective: To install and learn to operate the HP5890A capillary gas chromatograph and HP5895A ChemStation.

Status: The GC and ChemStation were set-up in January. The GC was equipped with a 15-meter Stabilwax-DB capillary column for nicotine analysis and a 30-meter DB-WAX capillary column for humectants, menthol and plasticizer analysis.

- B. Objective: To use capillary chromatography for nicotine in smoke analysis for ART cigarettes.

Status: The HP5890A capillary GC was set-up to perform nicotine analysis on ART smoke samples. Nicotine analysis is performed on a 15-meter x 0.25 mm ID x 0.25 micron film thickness Stabilwax-DB capillary column. The analysis time is 10 minutes per sample, including report generation on the ChemStation. Excellent agreement was shown between the Stabilwax-DB capillary column and the DB-5 wide-bore column previously used.

- C. Objective: To evaluate capillary versus wide-bore columns for nicotine analysis on ART smoke samples.

Status: A study was completed which showed excellent agreement between a 15-meter x 0.25 mm ID x 0.25 micron film thickness Stabilwax-DB capillary column and a 15-meter x 0.53 mm ID x 1.00 micron film thickness Stabilwax-DB wide-bore column. The wide-bore column was installed in an HP5880A packed column GC using HP injector port adapters and glass liners. A 5-meter fused silica guard column was installed before the analytical column which allowed the analysis time to be reduced from 40 to 20 minutes per sample. Although all ART smoke samples are currently being analyzed on a capillary GC, if a capillary GC is not available, nicotine can be analyzed on a packed column GC equipped with wide-bore columns.

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- D. Objective: To evaluate capillary versus wide-bore and packed columns for humectants, menthol and plasticizer analysis.

Status: Development work was started on the conversion of humectants, menthol and plasticizer analysis from packed and wide-bore columns to capillary columns. Packed columns lack the separating power required for complex samples, while wide-bore columns offer increased resolution but much longer analysis times. Preliminary work with a 30-meter x 0.25 mm ID x 0.25 micron film thickness DB-WAX capillary column on an HP5890A capillary GC indicates that analysis times of about 15 minutes per sample are possible and with higher resolution than with wide-bore columns.

The HP 5880A GC used for humectants analysis was modified to improve efficiency. The analysis is performed on a 30-meter x 0.53 mm ID DB-WAX wide-bore column with an analysis time of 45 minutes per sample. The installation of a five-meter x 0.53 mm ID fused silica guard column before the analytical column resulted in a decrease in analysis time to 25 minutes per sample.

- E. Objective: To replace the current Varian GC and VAX data processing system in the nicotine and water laboratory.

Status: Two HP 5890A gas chromatographs were placed into operation in July. The GCs are equipped with dual HP 7673 autosamplers, HP 3396 integrators, a capillary column system for nicotine analysis and a packed column system for water analysis. Laboratory technicians have been trained in the operation of the system.

Hewlett-Packard introduced the 3365 ChemStation software package in late 1989. This is a PC based product which offers full instrument control, integration of chromatograms and report generation. Two copies of the ChemStation software were purchased and one of the copies was installed in a Club 286 PC already located in the laboratory. This system will serve as a prototype for the entire laboratory automation system. CAD has been involved in interfacing the ChemStation to the VAX mainframe to transfer sample test results from the PC to the database.

- F. Objective: To select a replacement for the Filamatic system for filling sample tubes with extraction solution and develop a system to transfer solution from sample tubes to GC autosampler vials.

Status: A Gilson Model 222 sample changer was tested for sample tube filling and vialing operations. For sample tube filling, the Gilson is somewhat slower than the Filamatic, however, the dispensing is more accurate and reproducible. The Gilson system can dispense different volumes of solution by calling a program from the unit's keypad controller. The Gilson unit was also very efficient at transferring solution from sample tubes to GC autosampler vials. The programs supplied by Gilson were modified to ensure that there was no carryover or cross-contamination between samples during the transfer process.

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The Gilson system requires the use of a rubber septa for the 18 x 150 mm test tubes which were made of a softer rubber compound than those currently used. The West Company was able to supply a soft rubber septa which worked very well with the Gilson system.

II. TECHNICAL SUPPORT

- A. Objective: To investigate cyanide delivery from the four channel smoking procedure.

Status: In May, I was assigned to the Gas Phase Laboratory to try to bring hydrogen cyanide delivery for Monitor #25 cigarettes to within specification. During the next four months, I was able to learn the four channel smoking procedure and the influences of airflows and room conditions on the gas phase delivery of cigarettes. A series of steps were taken to try and isolate the cause of the problem which involved a close scrutiny of all aspects of the analysis: the smoking machines, valving, scrubbing towers, autoanalyzers, reagents, standards and smoking conditions. As a result of these efforts, the cyanide level for Monitor #25 increased significantly, but was still at the lower end of the calibrated limits.

- B. Objective: To set-up a capillary gas chromatograph for QA Product Audit.

Status: In February, I set-up an HP 5880A GC for Joe Garman in QA Product Audit to be used for nicotine analysis for ART smoke samples. This involved installing a 15-meter x 0.25 mm ID Stabilwax-DB capillary column, setting the GC operating parameters and optimizing the linear velocity and volumetric flow rate of carrier gas and setting the split ratio and septum purge flow rate. The response and chromatography from the capillary column was also checked and optimized.

- C. Objective: To provide technical and analytical support for Project Delta/Sigma and Project ART.

Status: We are continuing to analyze Delta/Sigma samples for nicotine, water and glycerin in smoke. Samples of extruded tobacco are also analyzed for PG and glycerin.

The nicotine and water laboratory continues to analyze a large number of ART smoke samples. Analysis of ART samples has now become a routine procedure.

III. MISCELLANEOUS

- A. Meetings and Seminars

Hewlett-Packard Capillary GC Seminar, Richmond, September.

J&W Scientific Seminar, Richmond, August.

Humidity Measurement Seminar, Richmond, October.

Dionex Ion Chromatography Seminar, Richmond, April.

Filtroha Users Group, Richmond, October.

Tobacco Chemists' Research Conference, Richmond, October.

B. Training Classes

Dionex Ion Chromatography Training Course, Atlanta, February.

Basic Techniques of Statistical Process Control, Richmond, October.

"Frontline Leadership"

- Basic Principles
- Giving Constructive Feedback
- Getting Your Ideas Across
- Recognizing Positive Results
- Dealing with Emotional Behavior
- Managing Effective Meetings
- Situational Leadership
- Establishing Performance Expectations

Selection Interviewing, in-house, July.

Salaried Discipline, in-house, June.

Performance Appraisal - Introduction, in-house, November.

C. Presentations and Reports

CTSD Project Meeting, "Nicotine Analysis," March.

CTSD Project Meeting, "Gas Phase Update," December.

Completion Report, "Determination of Nicotine in Smoke for Alkaloid Reduced Tobacco," Jane Y. Lewis, Robert A. Forte and Jeffrey A. Sampson, Accession No. 89-036, July 21, 1989.

D. Method

Draft Method "Nicotine in TPM by Capillary Gas Chromatography," submitted for Analytical Methods Manual, September.

E. Memos

Memo to Dr. Jane Y. Lewis, "Capillary Gas Chromatograph for QA," March 9.

Memo to Dr. Jane Y. Lewis, "Nicotine Analysis," March 13.

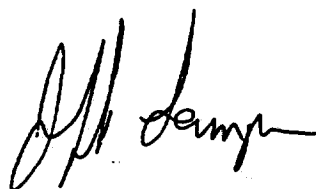
Memo to Dr. Jane Y. Lewis, "Proposed Systems for Nicotine and Water Laboratory," April 10.

Memo to Dr. Jane Y. Lewis, "Humectants Analysis," April 18.

Memo to Dr. Jane Y. Lewis, "Gas Phase Laboratory," June 9.

Memo to Mr. J. E. Wickham, "ISO Conditions in L-4217," June 15.

JAS:rad

A handwritten signature in dark ink, appearing to read "J. E. Wickham". The signature is fluid and cursive, with the first name "J" and last name "Wickham" clearly distinguishable.

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